भारतीय मानक
Indian Standard

IS 16212 : 2015

(Reaffirmed 2020)

सल्फोसल्फ्यूरान, तकनीकी — विशिष्टि

Sulfosulfuron, Technical — Specification

ICS 65.100.20

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June 2015 **Price Group 2**

Pesticides and Pesticides Residue Analysis Sectional Committee, FAD 01

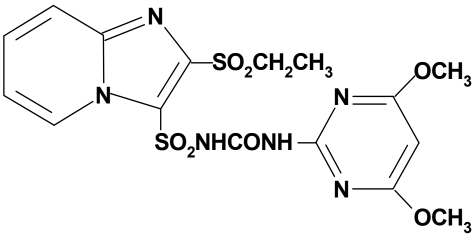
FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Pesticide and Pesticides Residue Analysis Sectional Committee had been approved by the Food and Agriculture Division Council.

Sulfosulfuron, technical is used in the preparation of herbicidal formulations for agricultural use.

Sulfosulfuron is the accepted common name by the International Organization for Standardization (ISO) for 1-(4,6-dimethoxypyrimidin-2-yl)-3-(2-ethylsulfonyl imidazo [1,2-a]pyridin-3-yl) sulfonylurea. The empirical formula, structural formula and the molecular mass are as indicated below:

Empirical Formula	Structural Formula	Molecular Mass
$C_{16}H_{18}N_6O_7S_2$		470.48



In the formulation of this standard, due consideration has been given to the provisions of *Insecticides Act*, 1968 and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under the *Insecticides Act* and Rules, wherever applicable.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

SULFOSULFURON, TECHNICAL — SPECIFICATION

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for sulfosulfuron, technical.

2 REFERENCES

The following standards contain provisions, which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
1070 : 1992	Reagent grade water (*third revision*)
6940 : 1982	Methods of test for pesticides and their formulations (*first revision*)
7601 : 1983	Fiberboard drums for general purposes (*first revision*)
8190 (Part 1) : 1988	Requirements for packing of pesticides: Part 1 solid pesticides (*second revision*)
10946 : 1996	Methods of sampling for technical grade pesticides (*first revision*)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of off-white, amorphous powder. It shall be free from extraneous impurities and hard lumps.

3.2 The material shall also comply with the requirements given in Table 1.

4 PACKING

The material shall be packed in polyethylene bags, closed suitably, and further inserted into fiberboard drums conforming to IS 7601. The containers shall also meet the general requirements given in IS 8190 (Part 1).

5 MARKING

5.1 The containers shall be securely closed, and the following information shall be marked legibly and indelibly on each container in addition to any other information, as is necessary under the *Insecticides Act*, 1968 and Rules framed thereunder:

a) Name of the material;
b) Name and address of the manufacturer;
c) Batch number;
d) Date of manufacture;
e) Date of expiry;
f) Net quantity;
g) Nominal Sulfosulfuron content, percent (*m/m*);
h) Cautionary notice worded as in the *Insecticides Act*, 1968 and Rules framed thereunder; and
j) Any other information required under the *Legal Metrology (Packaged Commodities) Rules*, 2011.

5.2 BIS Certification Marking

The product may also be marked with the Standard Mark.

Table 1 Requirements for Sulfosulfuron, Technical

(*Clause* 3.2)

Sl No.	**Characteristic**	**Requirements**	**Method of Test, Ref to**	
			Annex	Clause No. of IS 6940
(1)	(2)	(3)	(4)	(5)
i)	Sulfosulfuron content, percent by mass, *Min*	98.0	A	—
ii)	Melting point, °C	201 – 203	—	**6**
iii)	Acidity (as H_2SO_4), percent by mass, *Max*	0.50	—	**13.5.4**
iv)	Moisture content, percent by mass, *Max*	0.20	—	**4.1**

5.2.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986, and the Rules and Regulations made thereunder. The details of conditions, under which the licence for the use of the Standard Mark may be granted to manufacturers or producers, may be obtained from the Bureau of Indian Standards.

6 SAMPLING

Representative sample of the material shall be drawn as prescribed in IS 10946.

7 TESTS

7.1 Tests shall be carried out by the methods referred to in col 4 and col 5 of Table 1.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals and reagent grade water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[*Table* 1, *Sl No.* (i)]

DETERMINATION OF SULFOSULFURON CONTENT

A-1 PRINCIPLE

Sulfosulfuron content is determined by high performance liquid chromatography (HPLC) equipped with UV-VIS detector using internal standard technique.

A-2 APPARATUS

A-2.1 High Performance Liquid Chromatography System, equipped with UV/Vis detector and chromatographic software/printer plotter-cum-integrator or PC based data system. The suggested operative parameters are as follows, but can be changed, if necessary, provided standardization is done:

Column	:	Stainless steel, 250 mm × 4.6 µm i.d., packed with C-18 of 5 µm particle size
Temperature	:	Ambient
Mobile phase	:	Acetonitrile — 60 ml 0.008 7M KH_2PO_4 solution in water — 40 ml
Flow rate	:	1.0 ml/min
Detector	:	Ultraviolet detector (254 nm)
Injection volume	:	20 µl

A typical chromatogram of sulfosulfuron, technical with internal standard is given in Fig. 1

A-2.2 Analytical Balance, with an accuracy of 0.1 mg.

A-2.3 Ultrasonic Bath

A-2.3 Microlitre Syringe, 25 µl.

A-2.4 *p*H Meter

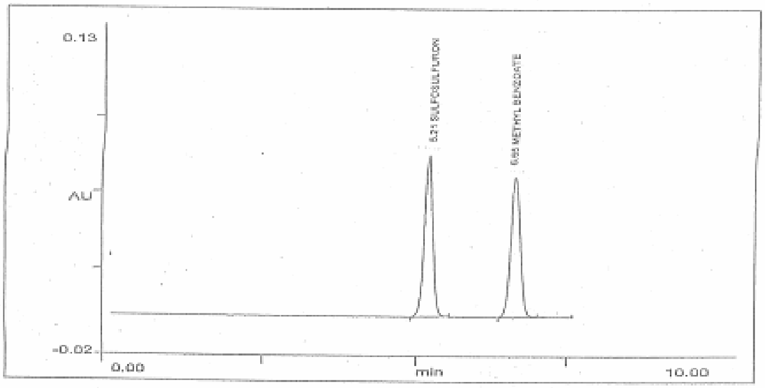


FIG. 1 TYPICAL CHROMATOGRAM

A-2.5 Standard Glassware

A-3 REAGENTS

A-3.1 Methyl Benzoate, internal standard**,** of known purity.

A-3.2 Sulfosulfuron Reference Standard, of known purity.

A-3.3 Potassium Dihydrogen Phosphate, AR grade, or equivalent – 0.008 7M KH_2PO_4 solution in water, prepared by dissolving 0.46 g of potassium dihydrogen phosphate in 400 ml of water and then adjusting the *p*H of this solution to 3.0 using a calibrated *p*H meter.

A-3.4 Water, AR grade, or equivalent.

A-3.5 Acetonitrile, AR grade, or equivalent.

A-3.6 Ortho Phosphoric Acid, AR grade, or equivalent, 85 percent.

A-4 PROCEDURE

A-4.1 Preparation of Internal Standard Solution

Weigh accurately 1.25 g of methyl benzoate into a 100 ml beaker. Transfer the contents quantitatively, into a clean 250 ml volumetric flask, by dissolving it in acetonitrile. Sonicate, and make up the volume upto the mark with acetonitrile. Shake well to homogenize.

A-4.2 Preparation of Standard Solution of Sulfosulfuron

Weigh accurately 75 mg of sulfosulfuron reference standard into a 100 ml volumetric flask. Add about 50 ml of acetonitrile, sonicate, and make up the volume up to the mark with acetonitrile. Shake well to homogenize. Pipette out 5 ml of this stock solution into a 50 ml volumetric flask. Add 5 ml of the internal standard solution to it. Make up to the mark with mobile phase. Shake well to homogenize. Call this as solution *C*.

NOTE — This solution is to be prepared fresh for every new set of analysis.

A-4.3 Preparation of Sample Solution

Weigh accurately 75 mg of the sample in to a 100 ml volumetric flask. Add 50 ml of acetonitrile, sonicate, and make up the volume up to the mark with acetonitrile. Shake well to homogenize. Pipette out 5 ml of this stock solution into a 50 ml volumetric flask. Add 5 ml of the internal standard solution to it. Make up the volume upto the mark with mobile phase. Shake well to homogenize. Call this as solution S_1. Prepare a duplicate sample solution in the same way. Call this as solution S_2.

A-5 ESTIMATION

Inject 20 µl of standard solution *C* (*see* **A-4.2**), until the area quotients of internal standard/standard of two successive chromatograms do not deviate from each other by more than 2 percent. Then use the following injection sequence:

$\ldots . C, S_1, S_1, C, S_2, S_2, C, \ldots .$

where

C = standard solution (*see* **A-4.2**); and

S_1, S_2 = sample solutions.

From the chromatograms of the standard solution, and sample solutions, measure the peak areas of the active ingredient and the internal standard, and calculate the percentage of the active ingredient as given in **A-6**. Take average values of the peak areas as indicated under **A-6**.

A-6 RETENTION TIME (GUIDE VALUES)

Sulfosulfuron	=	5.2 min
Methyl benzoate	=	6.7 min
Total run time	=	10 min

A-7 CALCULATION

Sulfosulfuron content (Calculated for S_1 and S_2 separately)**,** percent by mass

$$= \frac{M_1 \times A_1 \times A_3}{M_2 \times A_2 \times A_4} \times P$$

where

M_1 = mass of sulfosulfuron in standard solution, in mg;

M_2 = mass of sample taken for the test, in mg;

A_1 = average peak area of internal standard in the standard solution;

A_3 = average peak area of sulfosulfuron in the sample solution;

A_2 = average peak area of sulfosulfuron in the standard solution;

A_4 = Average peak area of internal standard in the sample solution; and

P = percent purity of sulfosulfuron standard.

NOTE — The average value of sulfosulfuron obtained for S_1 and S_2, must be reported.

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This Indian Standard has been developed from Doc No.: FAD 01 (1911).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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